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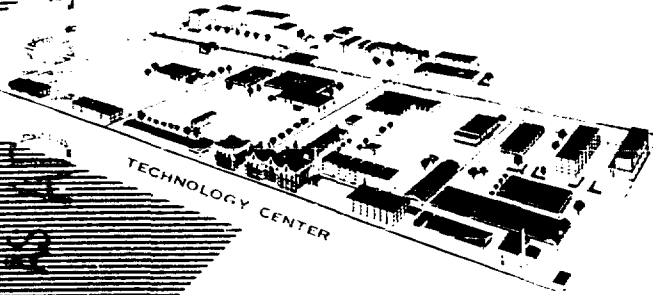
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(Summary Report)

ARMOUR RESEARCH FOUNDATION OF ILLINOIS INSTITUTE OF TECHNOLOGY



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A MECHANISM FOR STRESS-CORROSION EMBRITTLEMENT

Office of Naval Research  
Washington 25, D. C.

Contract No. Nonr-2602(00)

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ARMOUR RESEARCH FOUNDATION  
of  
Illinois Institute of Technology  
Technology Center  
Chicago 16, Illinois

Project No. ARF 2152  
Contract No. Nonr-2602(00)

A MECHANISM FOR STRESS-CORROSION EMBRITTLEMENT

Report No. ARF 2152-13  
(Summary Report)

August 1, 1959 - July 31, 1960

for

Office of Naval Research  
Department of the Navy  
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## A MECHANISM FOR STRESS-CORROSION EMBRITTLEMENT

### ABSTRACT

In order to bring the phenomenon of stress-corrosion cracking within the general framework of brittle fracture, the stress level for crack formation as a function of grain size has been investigated for a number of alloys. Stress-corrosion embrittlement of Al-4Cu in NaCl-H<sub>2</sub>O<sub>2</sub> solution at room temperature and Armco iron in boiling Ca(NO<sub>3</sub>)<sub>2</sub>-NH<sub>4</sub>NO<sub>3</sub> solution have been investigated. The stress for crack formation as a function of grain diameter for these alloys can be rectified in accordance with the Petch equation. From the slopes of data, surface energies of 160 and 32 ergs/cm<sup>2</sup> were calculated, respectively, for the Al-Cu alloy and Armco iron. These values of surface energy represent appreciable reduction from surface energy in a non-stress corroding medium and hence support the hypothesis that stress corrosion is indeed a brittle fracture phenomenon.

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## A MECHANISM FOR STRESS-CORROSION EMBRITTLEMENT

### I. INTRODUCTION

This is a summary report on Contract No. Nonr-2602(00) describing work done during the period August 1, 1959 to July 31, 1960. The purpose of this investigation was to analyze specific behavior characteristics of stress-corrosion embrittlement with the hope that this phenomenon could be brought within the present concepts of brittle fracture.

### II. THEORETICAL DISCUSSION

The phenomenon of fracture of a metal under static load in the presence of some particular liquid or gaseous media is known as stress-corrosion embrittlement. This has been interpreted classically to originate as a corrosion phenomenon, hence, the name.

In the last several years, principally by Stroh<sup>(1)</sup> and Petch,<sup>(2)</sup> theories have developed to correlate brittle fracture with dislocation theory. Essentially, these models postulate the formation of an embryo crack by the coalescence of dislocations in existence or produced by deformation processes. It is deduced that the relationship between fracture stress and grain size in brittle fracture is in the form:

$$\sigma_f = \sigma_o + k \left( \frac{1}{d} \right)^{1/2} \quad (1)$$

where  $\sigma_f$  = fracture stress

$d$  = average grain diameter

$\sigma_o$  and  $k$  are constants.

Petch<sup>(3)</sup> proposed a model of hydrogen embrittlement of mild steel which correlated embrittlement and reduction in surface energy,  $\gamma$ , with

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adsorbed hydrogen on the crack nucleus. He proposed that equation (1) be modified to:

$$\sigma_f = \sigma_o + 4 \left[ \frac{3 \mu \gamma}{\pi(1-\nu)} \right]^{1/2} \cdot \left[ \frac{1}{d} \right]^{1/2} \quad (2)$$

in which  $\gamma$  = surface energy of the crack

$\mu$  = modulus of rigidity

$\nu$  = Poisson's ratio

Thus, from the slope of the fracture stress-grain diameter it is possible to calculate the surface energy. Petch demonstrated that the surface energy of iron was reduced by hydrogen from 1600 to about 650 ergs/cm<sup>2</sup>.

That equation (2) is in essence correct has been demonstrated by Stroh<sup>(4)</sup> by the fact that surface energies calculated for iron, zinc, magnesium, and molybdenum are in good agreement with those derived by other methods.

Other workers have also postulated theories of brittle fracture which incorporate surface energy. Notable is that of Cottrell,<sup>(5)</sup> in which

$$\sigma_y k_y d^{-1/2} = \beta \mu \gamma$$

where  $\sigma_y$  = yield propagation stress

$k_y = \sigma_d l^{1/2}$  where  $\sigma_d$  is the unpinning stress and  $l$  is the distance from the source to the dislocation pileup

$\beta$  = constant

If the left side is greater than the right side, conditions are favorable for crack propagation and brittle fracture. Hence, a reduction in surface energy  $\gamma$  is conducive to brittle failure.

### III. PREVIOUS WORK

On the assumption that the Petch model for hydrogen embrittlement of mild steel could equally be adapted to the explanation of the stress corrosion embrittlement phenomenon, the present investigation was undertaken in July, 1958. During the initial year of work, the stress-corrosion cracking of 18-8 stainless

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steel in 42%  $\text{MgCl}_2$  at  $150^\circ\text{C}$  was investigated. In this investigation it was demonstrated that stress-corrosion cracking as a function of grain size obeyed the Petch type relationship. The surface energy calculated in these experiments was  $157 \text{ ergs/cm}^2$ , a marked reduction from  $1600 \text{ ergs/cm}^2$  for specimens in air.

During the initial experimental period of the current contract year, similar experiments were performed with a Mg-6Al alloy in a  $\text{NaCl-K}_2\text{CrO}_4$  solution at room temperature. In air, the surface energy,  $\gamma$ , was determined to be  $1737 \text{ ergs/cm}^2$ ; in stress-corroding medium,  $93 \text{ ergs/cm}^2$ .

The experimental investigation of the stress-corrosion embrittlement of 18-8 stainless steel has been completely discussed in the First Summary Report;<sup>(6)</sup> the stress corrosion investigation of the Mg-6Al alloy has been reported in a Technical Report<sup>(7)</sup> prepared for publication.

#### IV. PRESENT INVESTIGATION

During approximately the past nine months of the contract year, work was continued on the investigation of grain size dependence of stress-corrosion cracking. Inasmuch as the present theory has not been generally accepted by the scientific world, it was deemed necessary to accumulate an adequate amount of experimental data to justify the conclusion that indeed the surface energy  $\gamma$  was reduced in a stress-corrosion environment.

The stress-corrosion cracking of high-purity Al-4Cu alloy was determined in  $\text{NaCl-H}_2\text{O}_2$  solution at room temperature. The experimental data are summarized in Figure 1. From the line bounding data of "definite cracking" from "few cracks" a surface energy of  $160 \text{ ergs/cm}^2$  was calculated. The details of the experimental procedure have been described in Appendix A.

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Stress-corrosion cracking of a commercial grade of Armco iron was determined in a boiling solution of  $\text{Ca}(\text{NO}_3)_2 \cdot \text{NH}_4\text{NO}_3$ . From the slope of the line bounding data of "definite cracking" from "few cracks" presented in Figure 2, a surface energy of  $32 \text{ ergs/cm}^2$  was determined. Appendix B of this report gives the details of the investigation.

Limitations of experimental funds did not permit the determination of the surface energy for Al-4Cu and Armco iron to be made under non-stress corrosion conditions.

The stress-corrosion embrittlement data for stainless steel and the Mg-6Al alloy that have been discussed in previous reports are reproduced in the present report in Figures 3 and 4 for comparison.

#### V. CONCLUSIONS

As in the case of stainless steel and Mg-6Al, the behavior of Al-4Cu and Armco iron under stress corrosion conditions can be analyzed in terms of the Petch relationship with respect to grain size. The linearity of the data permits calculation of the surface energies from the slopes of the respective curves. Although surface energies for these alloys were not determined in a non-stress corroding medium, the extremely low values can leave no doubt that there has been a reduction in the surface energies.

The surface energies deduced are testimonial not only to the fact that the environment has reduced their magnitude but, more important, that the type of fracture encountered in stress-corrosion cracking is truly brittle. It will be recalled that Felbeck and Orowan<sup>(8)</sup> and Cottrell,<sup>(5)</sup> among others, have made the point that ordinary brittle fracture as produced by low temperatures is far from ideal--that is, a plastic work component in the energetics exists

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which outweighs the surface energy component by several orders of magnitude. Stress-corrosion cracking, therefore seems to fall into the category of an ideal brittle fracture.

The surface energy of iron has previously been determined<sup>(3)</sup> as approximately 1600 ergs/cm<sup>2</sup>. Taylor<sup>(9)</sup> has estimated the surface energies of several metals, but not aluminum. However, with the exception of potassium, rubidium, and cesium, no element has a calculated surface energy as low as that determined for Al-4Cu in a stress corroding environment. It seems logical that the calculated value of 160 ergs/cm<sup>2</sup> is less than the surface energy in a non-corrosive environment.

The stress-corrosion data for the Al-4Cu alloy and Armco iron are not as definitive as the previously reported data for stainless steel and Mg-6Al. In the latter systems, as demonstrated by Figures 3 and 4, there was a sharp discontinuity between "no cracking" and "definite cracking." However, for Al-4Cu and Armco iron the region of "few cracks" existed over a large range of stresses. In general, it would be difficult to establish a line bounding the region between "few cracks" and "no cracks." It was impossible to establish a region of "no cracks" for the Armco iron; specimens showed signs of cracking when bent even if the specimen had never been strained or subjected to the stress corroding medium.

VI. PERSONNEL AND LOGBOOKS

During the time interval in which the presently reported investigation of stress-corrosion embrittlement of Al-4Cu and Armco iron was performed, the following have been associated with the project:

Edward Coleman	Project Engineer
Rodney P. Elliott	Project Engineer
Robert H. Read	Supervisor
James Wright	Project Technician
Oswald Sanders	Project Technician

Pertinent data are recorded in ARF logbooks C-9128, C-1133, and C-1144.

Respectfully submitted,

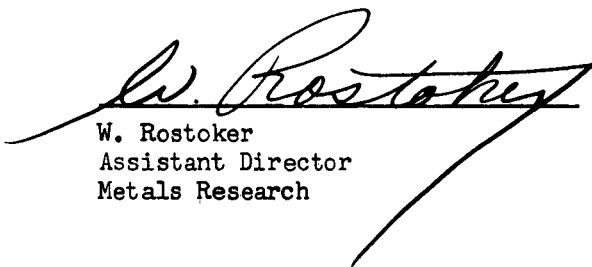
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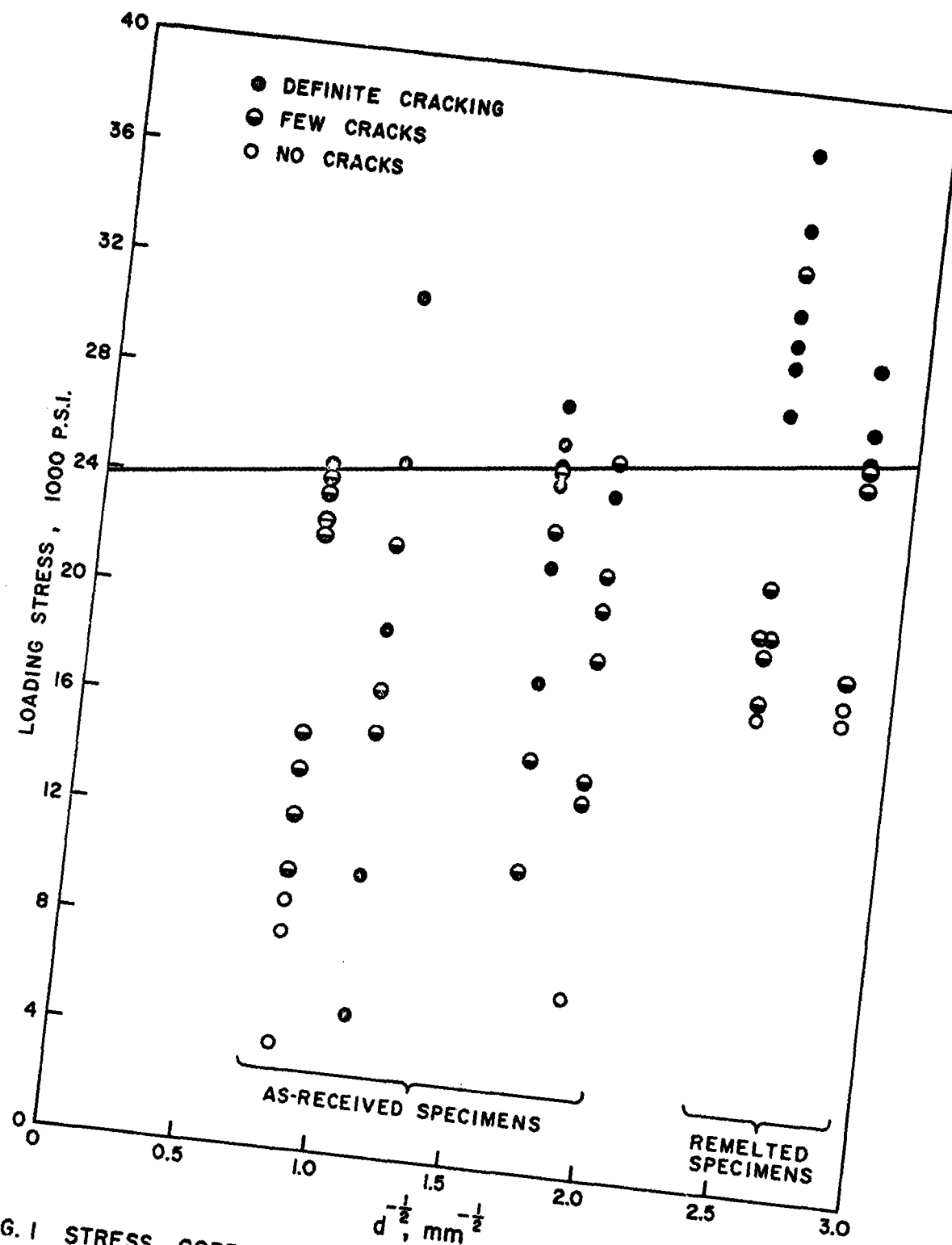


FIG. 1 STRESS CORROSION CRACKING OF Al-4%Cu ALLOY IN NaCl-H<sub>2</sub>O<sub>2</sub> SOLUTION

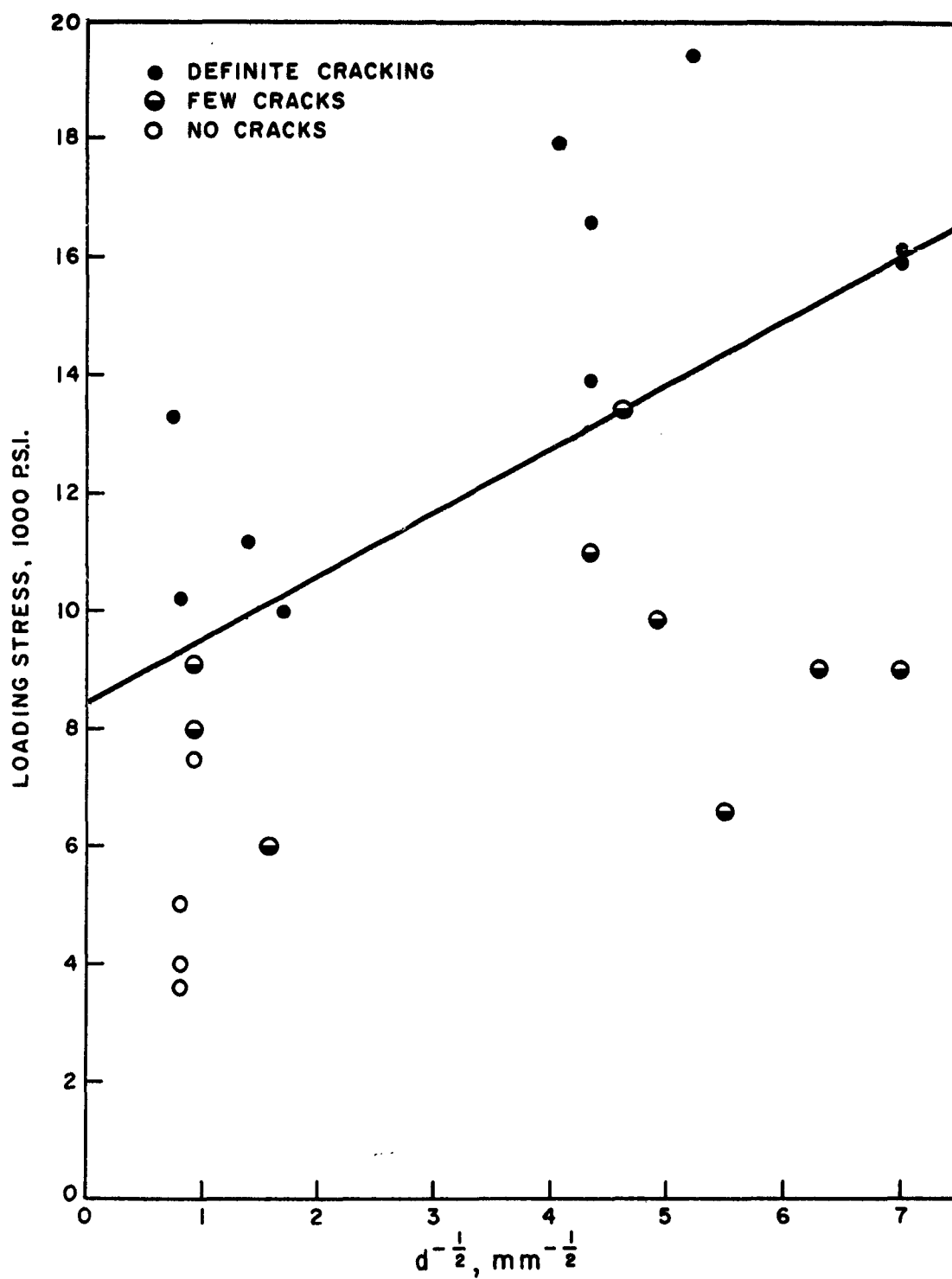


FIG. 2 - STRESS CORROSION CRACKING OF ARMCO IRON  
IN BOILING  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O} - (\text{NH}_4)\text{NO}_3$  SOLUTION.



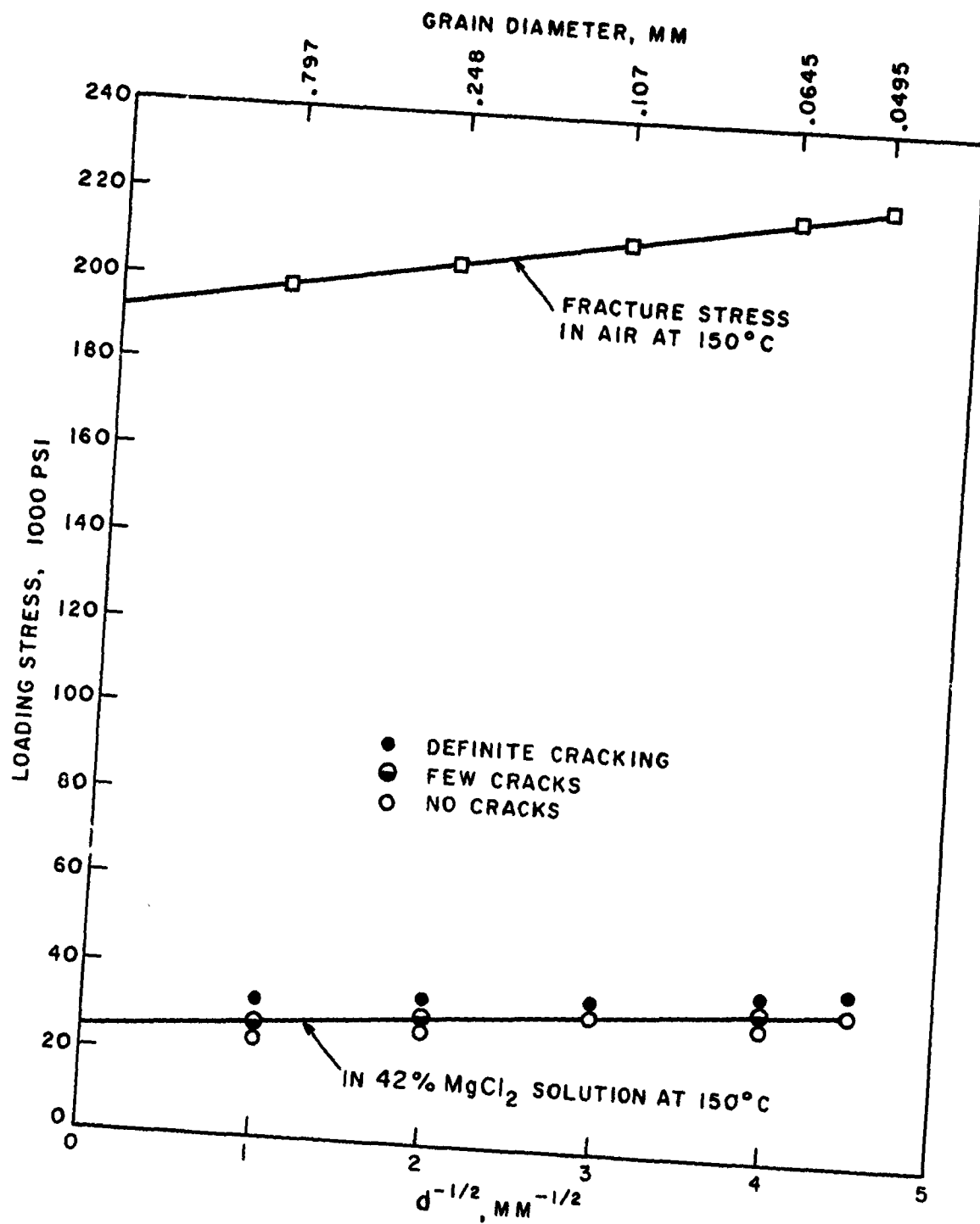


FIG. 3 - STRESS CORROSION OF STAINLESS STEEL.

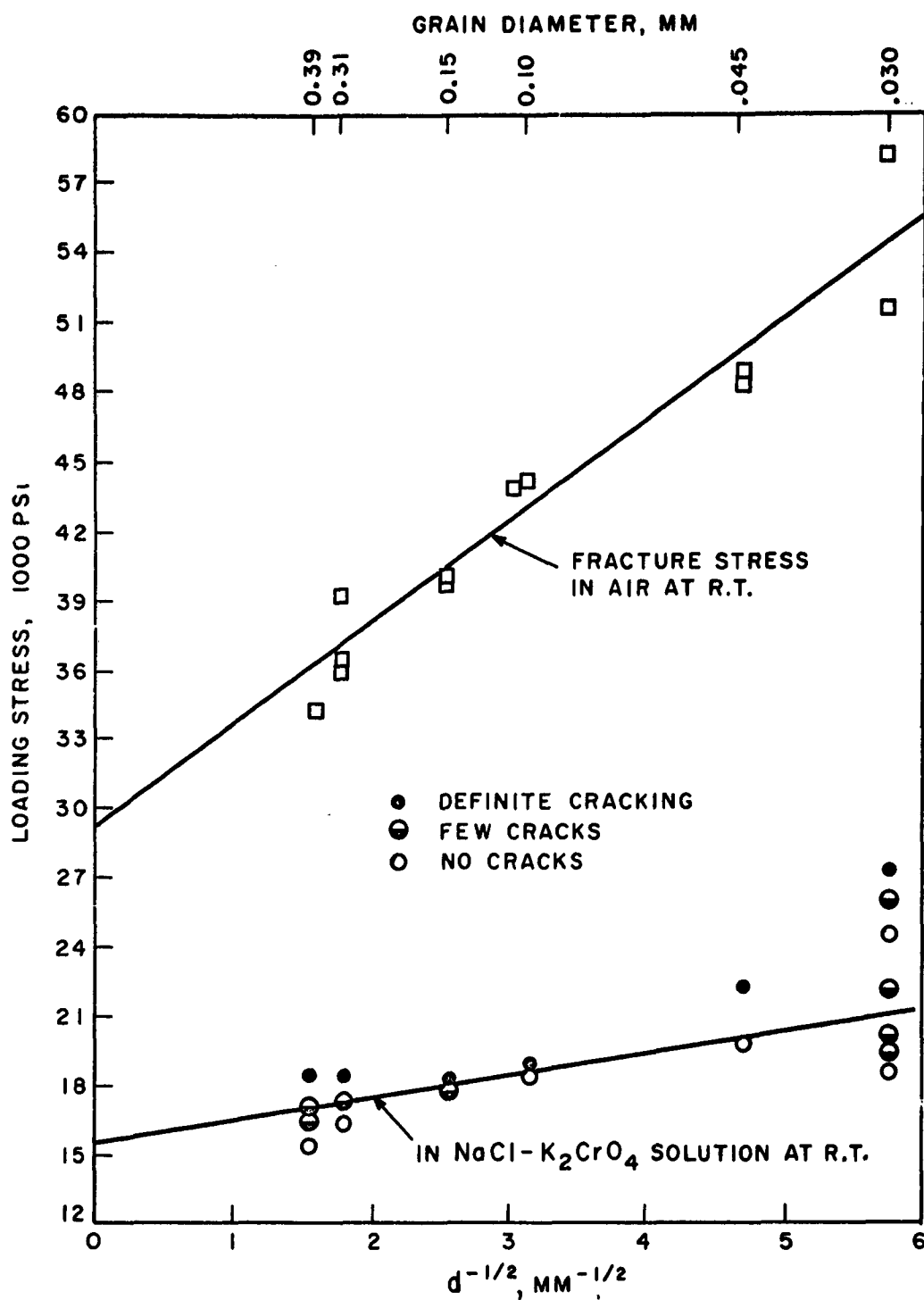


FIG. 4- STRESS CORROSION OF Mg-6Al.

## APPENDIX A

### STRESS-CORROSION EMBRITTLEMENT OF Al-4Cu

#### I. INTRODUCTION

This appendix describes the results of stress-corrosion embrittlement of a high-purity aluminum-4% copper alloy in a NaCl-H<sub>2</sub>O<sub>2</sub> solution at room temperature. From the slope of stress to produce cracking when plotted according to the Petch equation, a surface energy of 160 ergs/cm<sup>2</sup> has been calculated.

#### II. PREPARATION OF SPECIMENS

High-purity alloy of a nominal composition aluminum-4% copper was obtained from the Aluminum Company of America. This material was obtained as plate 1/2 in. x 8 in. x 12 in. and had the following analysis:

Cu	3.98 %
Fe	0.002
Si	0.003
Mg	0.001
other	0.000
Al	balance

The material as-received was annealed for two hours at 950°F prior to processing. Stock for flat tensile specimens was then produced by cold rolling. Tensile specimens were cut prior to the recrystallization anneal. By controlling the cold work, recrystallization temperature, and time at temperature, a variety of grain sizes between 0.37 and 1.50 mm was produced. The final recrystallization temperature was between 950° and 1000°F; specimens were water-quenched from the recrystallization temperature and subsequently aged at 360°F.

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Tensile specimens were cut from the cold-rolled stock on a Tensile-cut machine. The dimensions of the flat specimens produced are shown in Figure 5.

Grain sizes were determined by the Heyn intercept method, in which the grain boundaries are counted along an intercept line of known length

$$d = L/N$$

where d = the grain diameter

L = length of intercept line

N = number of grain boundaries

So that grain diameter data accumulated by such a technique are representative, the line length L was chosen so that approximately fifty grain boundaries were intersected.

Unfortunately, because of the high purity of the alloy, it was impossible to recrystallize into fine-grained specimens. To circumvent this, means were devised whereby the alloy was contaminated slightly with insoluble oxides, enabling finer grained specimens to be produced. Contamination was effected by melting in air and holding for approximately thirty minutes. The melt was then chill cast. It had originally been hoped that round specimens of a fine grained texture could be produced, but the alloy could not be cold swaged sufficiently to enable recrystallization to a fine grained structure without producing cold laps on the surface; flat tensile specimens were resorted to for the fine grain diameters. The recrystallization characteristics of the remelted alloy were investigated so that a minimum temperature and time could be selected to produce the smallest grain diameter. This was found to be five minutes at 930°F. Grain diameters of 0.143 and 0.162 mm were produced from the remelted Al-4Cu alloy.

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### III. STRESS-CORROSION TESTING PROCEDURES

The stress-corrosion embrittlement of Al-4Cu was investigated at room temperature in a NaCl-H<sub>2</sub>O<sub>2</sub> solution. The solution was always freshly prepared prior to testing so that the efficacy was unimpaired. The solution was prepared by adding 1 cc of 30% H<sub>2</sub>O<sub>2</sub> solution to 100 cc of a NaCl solution containing 53 gm NaCl/liter. All testing was performed on an Instron testing machine. The following procedure was used:

1. Assemble test specimen in fixture and connect to Instron.
2. Put stress corrosion solution in fixture.
3. Permit specimen to set in solution for five minutes.
4. Load to predetermined load at rate of approximately 1200 pounds per minute.
5. Immediately release load at same rate.

The cracks produced by such a procedure cannot be detected by the naked eye, nor can they be observed under the microscope. It was found necessary to bend the test specimen into an arc; the cracks that the arc produced are then detectable under the metallurgical microscope at magnifications in the range X100 to X500. It is extremely difficult to observe cracking in fine-grained specimens since the cracks that are produced are obviously only incipient failure cracks and are of the order of magnitude of the grain diameter in length.

The cracking of Al-4Cu alloy as a function of the grain diameter is summarized in the data presented in Figure 1.

### IV. CALCULATION OF SURFACE ENERGY

The stress-corrosion behavior of Al-4Cu has been presented in the text as Figure 1. From the line bounding the region of "definite cracking"

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from "few cracks" the following points were selected to construct the slope of the curve:

$$d^{-1/2} = 0$$

$$\sigma_0 = 23,700 \text{ psi} = 16.34 \times 10^8 \text{ ergs/cm}^2$$

$$d^{-1/2} = 3.0 \text{ mm}^{-1/2} = 9.486 \text{ cm}^{-1/2}$$

$$\sigma = 27,100 \text{ psi} = 18.69 \times 10^8 \text{ ergs/cm}^2$$

$$k_y = \frac{(18.69 - 16.34) 10^8}{9.486} = 2.477 \times 10^7$$

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$$\mu = \frac{E}{2(1 + \nu)}$$

$$E = 6.5 \times 10^6 \text{ psi} = 4.48 \times 10^{11} \text{ ergs/cm}^2$$

$$\nu = 0.34$$

$$\mu = \frac{4.48 \times 10^{11}}{2(1 + 0.34)} = 1.67 \times 10^{11}$$

- - - - -

$$\gamma = \frac{(k_y)^2 \pi (1 - \nu)}{48\mu}$$

$$= \frac{(2.477 \times 10^7)^2 3.1416 (1 - 0.34)}{48(1.67 \times 10^{11})}$$

$$\gamma = 1.6 \times 10^2 \text{ ergs/cm}^2$$

## APPENDIX B

### STRESS-CORROSION EMBRITTLEMENT OF ARMCO IRON

#### I. INTRODUCTION

This appendix describes the experimental procedure pertinent to the determination of the stress-corrosion embrittlement of Armco iron in a boiling  $\text{Ca}(\text{NO}_3)_2\text{-NH}_4\text{NO}_3$  solution. From the slope of the stress to produce cracking when plotted according to the Petch equation, a surface energy of  $32 \text{ ergs/cm}^2$  has been computed.

#### II. PREPARATION OF SPECIMENS

Armco Iron of the following chemical analysis was obtained for the preparation of stress-corrosion specimens:

C	0.02 %
Mn	0.03
P	0.09
S	0.023
Si	0.00
Cu	0.08
Fe	balance

This was obtained as cold-rolled slab  $1/2 \times 8 \times 18$  in. A second phase, probably manganese sulfide was evenly distributed throughout the iron. Since it was not preferentially distributed at grain boundaries, this effect can be ignored.

The nature of the iron-carbon equilibrium system complicated the development of a spectrum of grain sizes that was necessary in order to test the Petch relationship. Because of the carbon content, only a narrow temperature range could be used to grow the large grains and still maintain a single-phase structure. (It was deemed undesirable to anneal in a two-phase field

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since there would then be the possibility of producing a network of iron carbide around the grains. This could greatly influence the dislocation coalescence and crack formation.) By annealing in wet hydrogen for 3 to 120 hours (depending on specimen thickness) at 710°C, it was possible to decarburize the iron specimens and thereby permit recrystallization annealing over a much greater temperature region in order to develop the various grain sizes.

The slab Armco iron was cold rolled into flat sheet of various thicknesses from approximately 3/32 to 1/4 in. thick. The thicker specimens were designated for the larger grain diameters. Cold rolled plate was cut into tensile specimens as shown in Figure 5, and then annealed as previously described. Smaller grain diameters were produced by varying the annealing temperature. All specimens were given a terminal anneal at 720°C and water quenched.

It was difficult to produce very large grain diameters by varying the temperature; consequently strain-annealing techniques were employed. Tensile specimens that had been austenitized and quenched were strained from 5 to 9%, although 5% seemed to give better results. These were then slowly brought up to the terminal annealing temperature of 880°C from an initial temperature of 400°C by increasing the temperature 50°C every half hour. Specimens were held for two hours at 880°C, furnace cooled to 720° and held overnight, and water quenched.

Grain diameters from 0.04 to 2 mm were developed by equilibrium grain diameter annealing and the strain annealing techniques.

Grain diameter measurements were made by the Heyn intercept method described in Appendix A.

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### III. STRESS-CORROSION TESTING PROCEDURES

The stress-corrosion embrittlement of Armco iron was determined in a boiling solution of  $\text{Ca}(\text{NO}_3)_2 \cdot \text{NH}_4\text{NO}_3$ . The solution used was prepared by dissolving 860 gms  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  and 30 gms  $\text{NH}_4\text{NO}_3$  in water and diluting to one liter. Such a solution has a boiling point of approximately  $105^\circ\text{C}$ . All testing was done on an Instron test machine. The following test procedure was followed:

1. Load specimen in specially designed reflux condenser and assemble on Instron.
2. Put cold stress-corrosion solution in test fixture.
3. Heat stress-corrosion solution to boiling point (approximately three minutes are required).
4. Boil stress-corrosion solution around specimen for five minutes.
5. Load specimen to predetermined load at rate of approximately 1800 pounds per minute.
6. Immediately release load at same rate.

Stress-corrosion cracking was difficult to detect in Armco iron. It was necessary to bend the specimen into an arc to open the cracks. Magnifications of X100 to X500 were used to inspect the samples. Even with the bending technique, the specimen surface was severely pitted due to the manganese compounds. It was extremely difficult to detect cracks in very small grain diameter specimens since the pitting was of an order of magnitude larger than the cracks produced.

Observed stress-corrosion cracking data for Armco iron are summarized in Figure 2. From these data it has been possible to establish a line bounding the region "many cracks" from "few cracks." As can be seen, a region

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of "no cracks" has not been established except at large grain diameters (small values of  $d^{-1/2}$ ). In all probability, the presence of the manganese compound second phase is contributory to this phenomenon.

To evaluate the effect of the second phase, decarburized specimens that had not been subjected to the stress-corroding medium were bent and examined for cracks. Even specimens that had not been loaded showed cracks. This renders the observation of a few cracks for stress-corroded, fine-grain iron of no value and casts some doubt on the observation of many cracks at higher stresses. By processing untreated iron through the same thermal cycle as the decarburization treatment, it was established that decarburization was not contributory to this phenomenon. In order to establish a region of "no cracks" it would be necessary to fabricate specimens from high-purity melting stock.

#### IV. CALCULATION OF SURFACE ENERGY

The stress-corrosion behavior of Armco Iron has been presented in the text in Figure 2. From the line bounding the region of "definite cracking" from "few cracks" the following points were selected to construct the slope of the curve:

$$d^{-1/2} = 0$$

$$\sigma_0 = 8,400 \text{ psi} = 5.79 \times 10^8 \text{ ergs/cm}^2$$

$$d^{-1/2} = 7.0 \text{ mm}^{-1/2} = 22.13 \text{ cm}^{-1/2}$$

$$\sigma = 16,000 \text{ psi} = 11.03 \times 10^8 \text{ ergs/cm}^2$$

$$k_y = \frac{(11.03 - 5.79) \cdot 10^8}{22.13} = 2.367 \times 10^7$$

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$$\mu = \frac{E}{2(1+\nu)} \quad E = 30.4 \times 10^6 \text{ psi} = 20.96 \times 10^{11} \text{ ergs/cm}^2$$

$$\nu = 0.28$$

$$\mu = \frac{20.96 \times 10^{11}}{2(1 + 0.28)} = 8.188 \times 10^{11}$$

$$\gamma = \frac{(ky)^2 \pi (1-\nu)}{48\mu}$$

$$= \frac{(2.367 \times 10^7)^2 3.1416 (1 - 0.28)}{48(8.188 \times 10^{11})}$$

$$\gamma = 32 \text{ ergs/cm}^2$$

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